

# Seawater Immersion of GEM II Propellant

**Claude I. Merrill**  
**John D. O'Drobinak**

**AFRL/PRSP**  
**10 East Saturn Blvd.**  
**Edwards AFB CA 93524-7680**

**February 1999**

**Final Report**

**19990427 085**

---

**APPROVED FOR PUBLIC RELEASE; DISTRIBUTION UNLIMITED.**

---



**AIR FORCE RESEARCH LABORATORY**  
**AIR FORCE MATERIEL COMMAND**  
**EDWARDS AIR FORCE BASE CA 93524-7048**

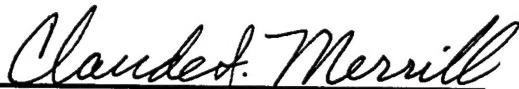
## NOTICE

When U.S. Government drawings, specifications, or other data are used for any purpose other than a definitely related Government procurement operation, the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data, is not to be regarded by implication or otherwise, or in any way licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use or sell any patented invention that may be related thereto.

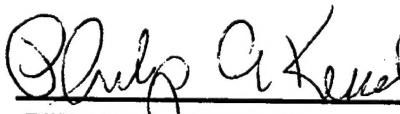
## FOREWORD

The work reported in this final report was performed for the 45th Space Wing/SESM, Patrick AFB, FL under JON: PATR0084. The principal investigators at the Air Force Research Laboratory, Propulsion Directorate, Edwards Facility, Edwards AFB CA 93524-7680 were Dr. Claude I. Merrill and John D. O'Drobinak.

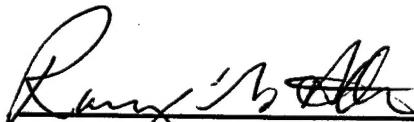
This report has been reviewed and is approved for release and distribution in accordance with the distribution statement on the cover and on the SF Form 298.



CLAUDE I. MERRILL  
Project Manager



PHILIP A. KESSEL  
Technical Advisor  
Propulsion Sciences and Advanced  
Concepts Division



RANNEY G. ADAMS  
Public Affairs Director

**REPORT DOCUMENTATION PAGE**
**Form Approved  
OMB No 0704-0188**

Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0740-0188), Washington DC 20503.

1. AGENCY USE ONLY (LEAVE BLANK)	2. REPORT DATE	3. REPORT TYPE AND DATES COVERED	
February 1999		Final 1 May 1997 – 30 Sep 1998	
4. TITLE AND SUBTITLE		5. FUNDING NUMBERS	
<b>Seawater Immersion of GEM II Propellant</b>		C: PE: PR: PATR TA: 0084	
6. AUTHOR(S) Claude I. Merrill and John D. O'Drobinak		8. PERFORMING ORGANIZATION REPORT NUMBER	
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Air Force Research Laboratory (AFMC) AFRL/PRSP 10 East Saturn Blvd. Edwards AFB CA 93524-7680		AFRL-PR-ED-TR-1998-0034	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) 45 Space Wing/SESM 1201 Edward H. White II Street, MS 7300 Patrick AFB FL 32925-3239		10. SPONSORING/MONITORING AGENCY REPORT NUMBER	
11. SUPPLEMENTARY NOTES			
<b>COSATI CODE(S):</b> 12a. DISTRIBUTION/AVAILABILITY STATEMENT Approved for Public Release; Distribution is Unlimited			
12b. DISTRIBUTION CODE A			
13. ABSTRACT (MAXIMUM 200 WORDS)			
Experimental data has shown that safety risks associated with GEM propellant from the 17 January 1997 Delta II flight failure diminish with time. It was shown that AP leached out of GEM propellant exposed to seawater at a logarithmic rate with a slope of 0.44 (% AP lost/week aged in seawater) and intercepts that depend on sample size. Friction and impact data on dried aged propellant samples showed no increased burning hazard compared with propellant not exposed to water. With increased seawater aging, the sensitivity of the outer surfaces of the dried propellant samples gradually decreased. The outer surface of wet propellant samples were significantly less sensitive to friction and impact than virgin propellant. Centers of wet propellant samples were found to be less sensitive than propellant not exposed to water, but definitely more sensitive than wet propellant sample surfaces. In fuel fires, initially, no differences could be observed between burning fresh propellant and dried, aged propellant. With increased seawater aging and resultant AP loss, the flame intensity of these burning aged propellants gradually decreased. During the early stages of aging, all dry samples ignited immediately and burning spread over all exposed surfaces. As seawater-aging time increased, flame spreading to all exposed surfaces gradually decreased. Finally, it was demonstrated that all wet aged samples experienced ignition delays. Ignition delays were directly related to water immersion time. These samples only burned on one face leaving an oxidizer poor rubber shell. A point was reached with the 1- and 2-in. propellant cubes where it became essentially impossible to obtain sustained combustion under the conditions used in the Fire Test.			
14. SUBJECT TERMS		15. NUMBER OF PAGES	
seawater aging; GEM II propellant; aging study; safety; ammonium perchlorate depletion; AP depletion		25	
17. SECURITY CLASSIFICATION OF REPORT		16. PRICE CODE	
Unclassified			
18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT	
Unclassified	Unclassified	SAR	

## TABLE OF CONTENTS

	<u>Page</u>
<b>SUMMARY</b>	1
<b>INTRODUCTION</b>	2
<b>EXPERIMENTAL</b>	2
<b>Sea Water Aging</b>	2
<b>Fire Tests</b>	2
<b>Safety Tests</b>	7
<b>RESULTS</b>	7
<b>CONCLUSIONS</b>	21

## LIST OF FIGURES

<u>Figure</u>		<u>Page</u>
1	Temperature Controlled Building in Which Aging Study was Conducted	3
2	Swimming Pool Inside Aging Study Building Showing the Cover Tarp Partially Removed with String Hanging over the Sides of the Pool Which Are Connected to Baskets Containing Propellants	4
3	Circulation Pump with Hoses Connected to the Swimming Pool	5
4	Two Baskets Containing Propellant Samples on Bottom of Pool with the Basket on the Left Containing Samples Buried in Sand	6
5	AP Depletion Rates for GEM Propellant Aged in Simulated Seawater	7
6	AP Depletion for GEM Propellant Aged in Pacific Ocean Seawater	9
7	Appearance of Blisters on GEM Propellant Aging in Pacific Seawater	10
8	Cutting into the Blister on GEM Propellant Cube Aged in Pacific Seawater	11
9	Appearance of Blister on GEM Propellant After it had been Almost Completely Cut Through	11
10	Under Surface of Cutaway Blister on GEM Propellant Showing Underside Surface of Blister and Water Filled Void Beneath it	12
11	Water Removed from Void Underneath Cutoff Blister on GEM Propellant Showing its Surface	12
12	Relative Swelling Rates of Wet Aged GEM Propellant	13
13	Representative Appearance of Wet and Dry GEM Propellant Cubes that Were Aged in Pacific Seawater	14
14	Differential Scanning Calorimeter (DSC) Thermogram of Ammonium Perchlorate (AP)	15
15	Differential Scanning Calorimeter (DSC) Thermogram of White Crystalline Spots Located on Surfaces of Dried Cubes	15
16	Post Fire Test Results on 1-in. GEM Propellant Cubes that Were Aged from 15.12 to 43.43 Weeks in Pacific Seawater	17
17	Shell Left by a 2-in Wet GEM Propellant Cube Aged for 43.43 Weeks in Pacific Seawater After a Fire Test in Which It Was Adjacent to a 2-in. Dry Cube Aged for the Same Period	19

## LIST OF TABLES

<u>Table</u>		<u>Page</u>
1	Ammonium Perchlorate Percent Depletion/ Loss Data	8
2	Projected AP Depletion Half Lives for GEM Propellant Aged in Seawater	8
3	Friction and Impact Test Results of Aged GEM Propellant	16
4	Effect of Aging Time and Sample Size and State on Fire Test Results for Tests 1-35	18
5	Effect of Aging Time and Sample Size and State on Fire Test Results for Tests 36-58	20

## SUMMARY

This program was initiated in response to the Delta II flight failure of 17 January 1997 at Cape Canaveral. There were concerns that propellant which broke up and fell into the ocean might eventually wash ashore and might present fire and explosive hazards. To explore these possible hazards, the effects of seawater on GEM II propellant were determined as a function of time by subjecting seawater aging GEM II propellant to fire, friction and impact tests.

GEM II propellant was aged in seawater, which was contained in a large portable swimming pool, for a period of 58 weeks at temperatures that varied from 69 to 74°F. Samples, which were representative of actual propellant fragments, were aged as 1-in., 2-in., 4-in. and 15-in. cubes. To approximate the effect of tidal action burial of real propellant fragments in the ocean, some of the cubes were buried in sand. Aging data showed that ammonium perchlorate (AP) leached out of the different propellant cubes at a logarithmic rate as exemplified by parallel lines that had a slope of 0.44 (%AP lost/week aged in seawater). The intercepts of these parallel lines depended on sample size. AP is leached out of the propellant very slowly. This is aptly demonstrated by looking at the times it takes for half of the AP to be leached out of the different sized propellant cubes. The 1-, 2- and 4-in. cubes require 0.8, 3.5 and 14.4 years, respectively.

Drying propellant cubes after aging in seawater led to the appearance of white spots on the surfaces of the cubes. Differential Scanning Calorimetric data on the white spot material, as well as on a sample of authentic AP, showed that the white spots were AP.

Standard impact and friction tests were run on both wet and dried seawater aged propellant cubes using an Olin Mathieson Model 7 Drop Weight Tester and a Julius Peters Model 21 Friction Tester. Impact data showed that wet aged propellant is significantly less reactive than either dry virgin propellant, which has not been aged, or dried aged propellant. This is particularly true for the exposed surface areas. The center areas of these wet cubes are not as insensitive to impact as the outer skin or surface areas, but they are very definitely less sensitive than any corresponding dry aged cube. Also, the centers of wet 1-in. cubes are less sensitive than those of correspondingly aged 2-in. cubes since more AP leaches out of the 1-in. cube because of its more favorable surface-area-to-weight ratio. Finally, after about 50% of the AP leached out of 1-in. cubes, which were then dried; their surface skins are about as insensitive as the surface skins of any wet aged cubes. Friction data shows that the wet outer surface skins of aged cubes are much less sensitive than their centers. Regardless, centers of these wet cubes are similar in sensitivity to that of dried and aged cubes. This friction data, like the impact data, shows that after about 50% of the AP has leached out of the 1-in. cubes, which were then dried, their surface skins are as insensitive as the surfaces of any aged wet cubes.

Fired tests were run for both wet and dry propellant cubes that had been aged in seawater together with a dry unaged control cubes. The seawater soaked, but dry, cubes behaved very much like a dry unaged propellant. The cubes ignited rapidly and the flame front quickly spread over the entire surface of the cubes. After about 15 weeks aging in seawater, the dry 1-in. cubes started experiencing gradually increasing cube ignition delay with their flame fronts spread more slowly over all the exposed surfaces. By this time enough AP had been leached out of the aged samples so that a portion of the samples acted as though they were insulators. Fire tests on the wet aged cubes required longer heating times before the cubes could sustain combustion. Once combustion was sustained, burning occurred on only one surface or side. Increased aging (and resultant increased AP leaching) made sustained combustion increasingly difficult and at times impossible under the conditions of the tests (engulfing isoctane fire). Initially, a relatively low level isoctane flame was used to provide relatively long heating periods before sample ignition occurred so that a more violent behavior might possibly be obtained as compared to a totally engulfing fire. Quiet burning was the only action observed with the propellant samples.

Generally, with increased aging time and resultant AP leaching loss, all wet propellant samples become increasingly less sensitive to friction, impact and burning. Corresponding dry samples decrease in sensitivity at a significantly slower rate.

## INTRODUCTION

The 17 January 1997 Delta II flight failure at Cape Canaveral raised the question of public safety from the solid propellant that had fallen into the Atlantic Ocean. With this failure, approximately 200,000 pounds of HTPB solid propellant were released. While large amounts of propellant were consumed in burning fragments and ground impact explosions, considerable amounts of unburned propellant fell onto the land and into the Atlantic Ocean. It was quickly found that propellant attacked by seawater became mushy, and ammonium perchlorate (AP) crystallized on propellant surfaces if it was allowed to dry. Concerns were raised that propellant washing ashore might present fire and, particularly, explosive hazards. In response to these concerns, this program was initiated to investigate the effects of seawater on GEM II propellant as a function of time and its activity when burned, impacted or subjected to friction. Based on the Delta II accident investigation, propellant samples, representative of actual fragment sizes, would be aged. Propellant cubes (1-in., 2-in and 4-in. cubes) would demonstrate the validity of geometric scalability. Aging was conducted in Pacific Ocean seawater under ambient conditions.

## EXPERIMENTAL

### Sea Water Aging

GEM II propellant samples were aged in a portable 25 x 13.5 x 4.5-foot swimming pool filled with Pacific Ocean seawater. The pool contained 10,500 gallons of seawater that had been previously sand filtered. Fitted with a circulation pump, it was housed in a temperature controlled building (Figs. 1-3). Water temperature usually varied from about 69 to 74°F. Propellant samples were placed in open plastic crate-type boxes that had string or rope attached for easy removal at periodic intervals for sample retrieval (Fig. 4). The 15-in. cubes were loosely held with plastic webbing. Some 1- and 2-in. propellant samples, buried in sand, were placed in open plastic crates. These crates were lined with porous mesh plastic sheeting to keep sand from flowing out of the crates and exposing the samples. For comparison some 1-in. GEM II propellant cubes were aged in deionized water.

Pacific Ocean seawater was used in this study since analysis showed no significant differences in mineral content between Pacific Ocean seawater and Cape Canaveral seawater. Pacific Ocean seawater was readily available at a shorter distance and lower cost. At the beginning of the program simulated seawater was used to age 1-in. and 2-in. propellant cubes to check the validity of the program's experimental approach.

### Fire Tests

The 1-, 2- and 4-in. propellant cubes were burned in a 1-in. deep 8- x 12-in. stainless steel tray filled with sand. An electric match was used to ignite the samples. Before a propellant sample was placed on the sand, the sand was saturated with isoctane fuel. Test videos recorded times-to-ignition and burning vigor. The dry cubes experienced no ignition problems. The wet cubes were another matter. For a while two test modifications were used successfully. Initially, the isoctane fuel was replaced with isopropanol fuel. Because of isopropanol's high flash point and the position of the electric match, isoctane was used in the vicinity of the electric match. When ignition could not be obtained with isopropanol, a dry cube was placed within a  $\frac{1}{4}$ -in. of a wet cube.

The 15-in. propellant cubes were burned in a 4-in. deep, 4- x 4-ft. stainless steel tray filled with sand. Again, using an electric match about 1-in. above the fuel-saturated sand the fire was ignited remotely. Because of their great weight (both dry and wet), bricks were placed on the bottom of the tray spaced to support the cubes near their corners while they were flush with the top of the sand filling. About 5 gallons of isopropanol was used to test dry cubes and about 8 gallons used to test wet samples.

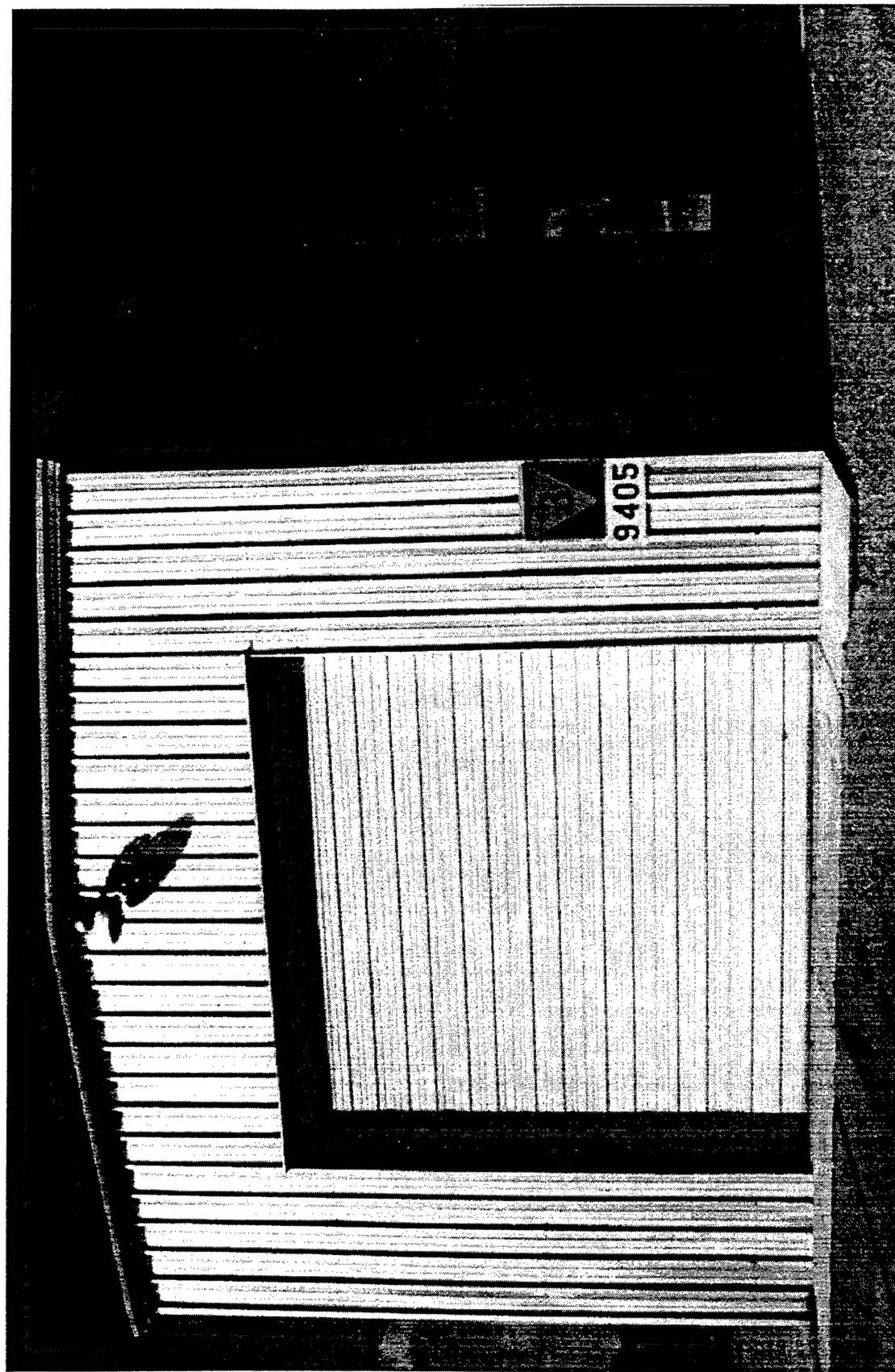


Figure 1. Temperature Controlled Building in Which Aging Study was Conducted

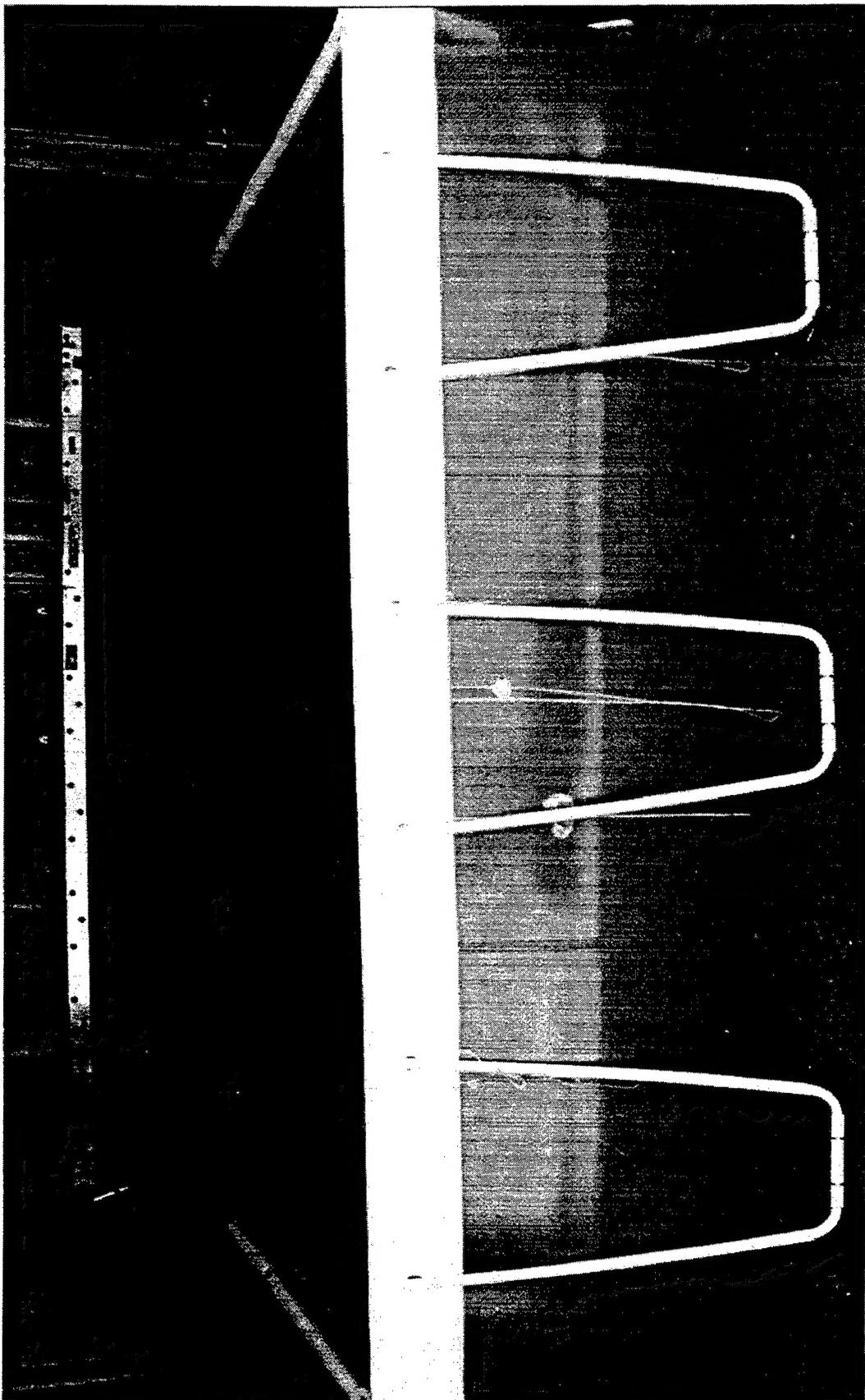


Figure 2. Swimming Pool Inside Aging Study Building Showing the Cover Tarp Partially Removed with String Hanging over the Sides of the Pool Which Are Connected to Baskets Containing Propellants

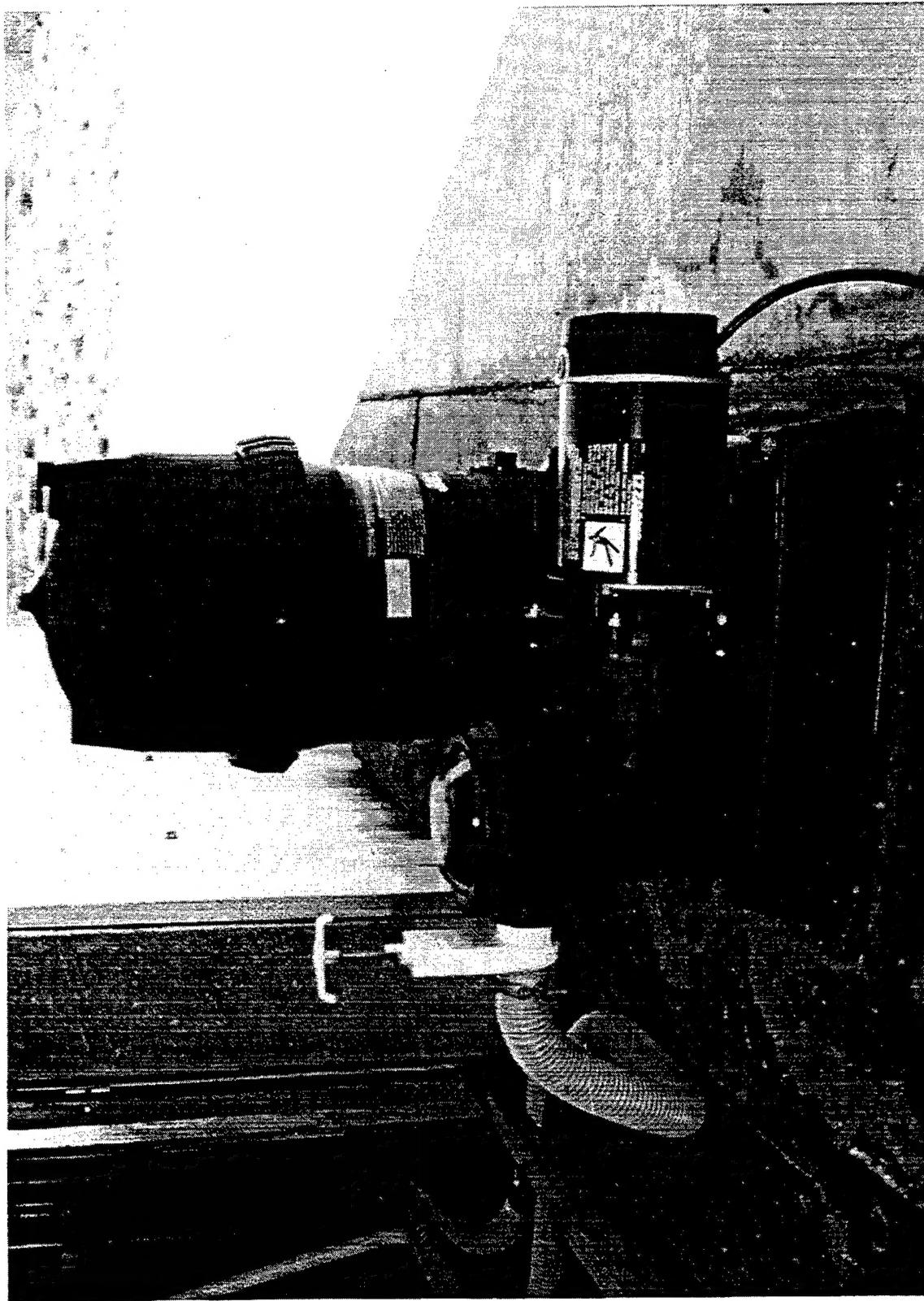
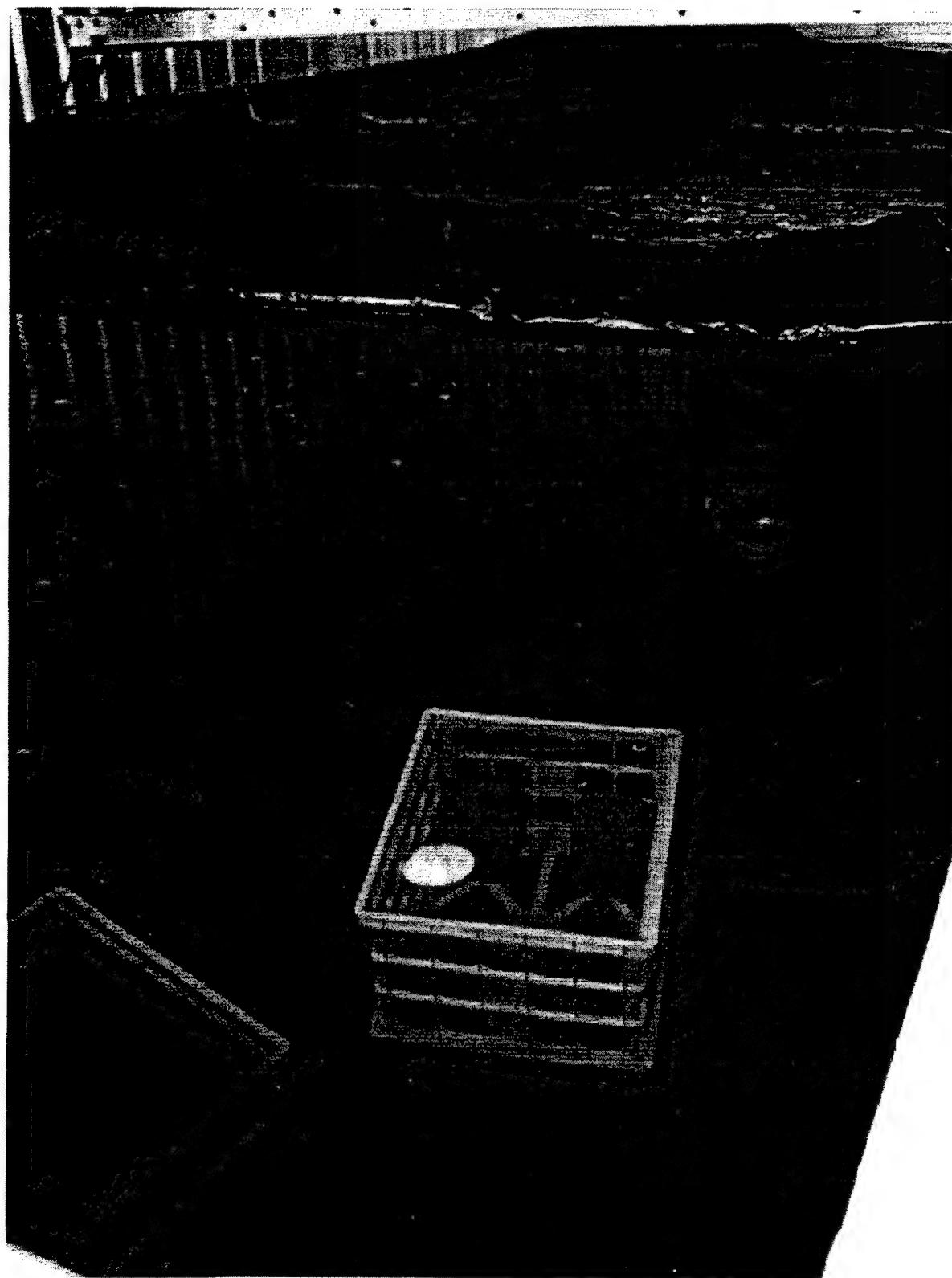


Figure 3. Circulation Pump with Hoses Connected to the Swimming Pool



**Figure 4. Two Baskets Containing Propellant Samples on Bottom of Pool with the Basket on the Left Containing Samples Buried in Sand**

## Safety Tests

Friction and impact tests were run on both dry and wet aged cubes. Cube test areas of prime interest were the outer skin surfaces. The center areas of the cubes were also tested. Friction test data was obtained using a Julius Peters, Model 21 Friction Tester. Impact test data was obtained using an Olin Mathieson, Model 7 Drop Weight Tester.

## RESULTS

Prior to the principal aging study, a preliminary aging study was initiated using samples obtained from a small propellant mix of Delta GEM propellant. Both 1- and 2-in. samples were aged in a 250-gallon container of simulated seawater. This preliminary study was performed to obtain aging data as soon as possible to validate the proposed program techniques and approaches before a larger 150-gallon propellant batch was made at the Naval Air Weapons Center (NAWC) for use in the primary study.

Oxidizer or ammonium perchlorate (AP) depletion rates were determined for 1- and 2-in. propellant cubes aged in simulated seawater (SS). Prior to aging, all cube surfaces were freshly machined. Results are shown graphically in Figure 5. Both 1- and 2-in. cubes lost AP in an orderly manner with respect to size and time. The data showed that the proposed aging plan for the different sized propellant cubes was valid.

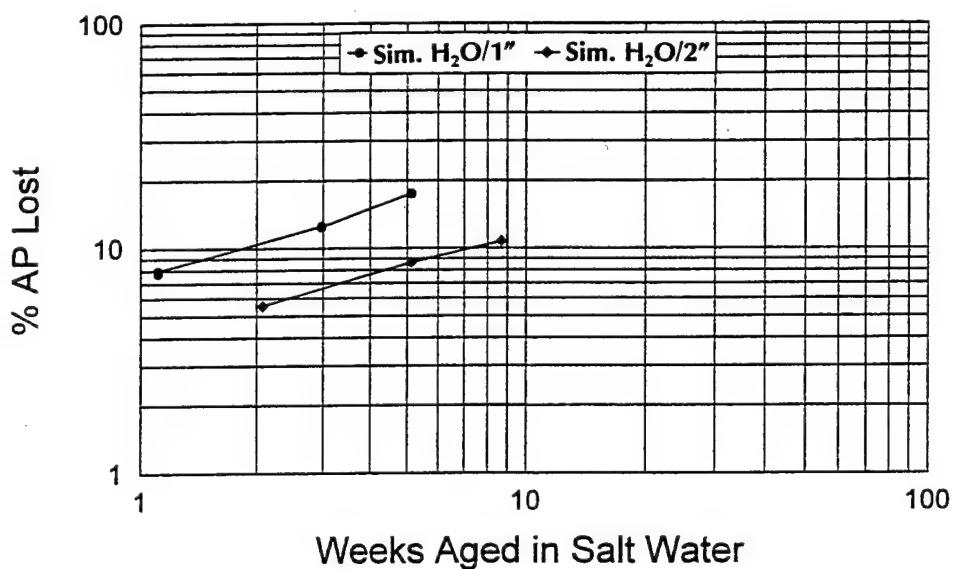


Figure 5. AP Depletion Rates for GEM Propellant Aged in Simulated Seawater

A 150-gallon mix of Delta GEM propellant was made at the NAWC, China Lake, CA. Propellant bars with square cross sectional areas exceeding 1-, 2- and 4-in.<sup>2</sup> were cast and cured as well as 15-in. cubes. Propellant bars were cut into 1-, 2- and 4-in. cubes. All binder rich surfaces resulting from casting operations were removed. Removal of binder rich cube surfaces was intended to minimize differences between samples and accident produced fractured propellant in AP leaching rate during seawater aging. After cutting and surface preparation operations, the cubes were weighed and measured prior to being immersed in a Pacific Ocean seawater filled pool. Some of the 1- and 2-in. cubes were buried in sand contained in plastic crates prior to being immersed in the pool. These particular samples would simulate the effect of propellant buried under sand by tidal action of the ocean. The aging study was conducted for 58 weeks. Aging data is presented in Table 1 and shown graphically in Figure 6. It shows that AP leaches out of GEM propellant at a logarithmic rate with a slope of 0.44 (%AP lost/week aged in seawater) and with

intercepts dependent on sample size. It is interesting to note that the sand buried samples (1- and 2-in. cubes) appear to lose AP at the same rate as open water exposed samples. This was surprising because one would expect that the depletion rate would be less for the buried samples because of sand interference with diffusion. Some important observations shed some light on this matter. All of the water exposed propellant cubes were covered with slime. The slime was probably caused by microorganisms present in the Pacific Ocean seawater. (This slime was also observed in preliminary propellant cube work using water from Cape Canaveral. This showed the universal presence of microorganisms in seawater.) The cubes that were buried in sand were never covered with slime. Regardless, the depletion rates for both slime covered and sand-buried cubes were identical. This shows that any effects that might possibly be attributed to either the presence of slime or sand do not exist.

Table 2 lists projected times covering AP half lives from the 1-in., 2-in. and 4-in. propellant cubes while immersed in seawater. One can readily see that the AP depletion rate is very slow. This is evidenced by the range of values for 50% AP depletion where the sample's weight-to-surface-area is manifested. Thus Delta GEM propellant pieces lying on the bottom of Cape Canaveral of comparable size to this study's 4-in. propellant cubes will be around for over 14 years before half of their AP is depleted. Smaller propellant pieces will take less time for comparable dissolution and larger pieces will take more time for comparable dissolution.

**Table 1. Ammonium Perchlorate Percent Depletion/Loss Data**

Time <sup>a</sup>	1" Cube	2" Cube	4" Cube	15" Cube	1" Cube/Sand <sup>b</sup>	2" Cube/Sand <sup>b</sup>	1" Cube/Dion <sup>c</sup>
2.00	12.20, 13.50	6.35					
3.40	16.34, 15.86						
4.00		8.51	4.33				
4.43							17.58
6.97	21.74, 20.76						
8.00		12.44	6.33				
9.97					26.13, 27.13	13.57	
11.00							28.89, 28.12
15.12	31.72, 31.76	17.31					
17.14			8.89				
17.34							34.62, 34.24
19.00					33.66, 33.33	18.25	
27.12							42.65, 42.94
32.00							46.62, 46.15
33.00	42.24, 41.83	22.79, 22.22			40.48	23.18, 21.98	
34.29					3.69		
43.43	49.32, 48.87	25.39, 25.54					
53.12	53.08, 51.88	27.70, 27.56					
58.00	56.27, 54.785	30.91, 29.67	15.30, 15.34		4.02		
	7.77						

a. Time samples aged in seawater as weeks.

c. Cubes aged in deionized water.

b. During aging cubes were buried in sand.

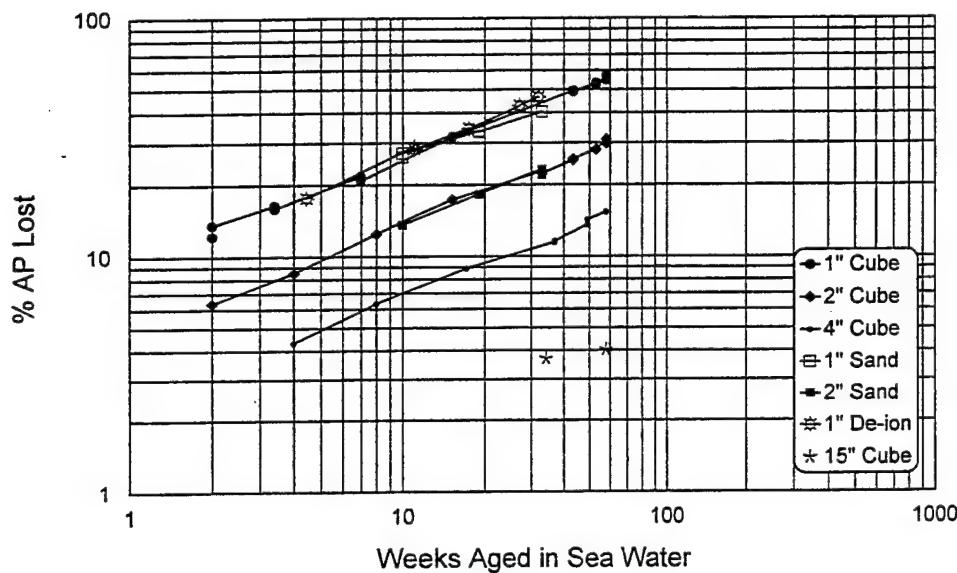
**Table 2. Projected AP Depletion Half Lives for GEM Propellant Aged in Seawater**

Sample Size	Half Life <sup>a</sup>
1" Cube	43 <sup>b</sup> /0.83 <sup>c</sup>
2" Cube	180 <sup>b</sup> /3.46 <sup>c</sup>
4" Cube	750 <sup>b</sup> /14.42 <sup>c</sup>

a. Time for half of AP to be depleted.

b. Time in weeks

c. Time in years



**Figure 6. AP Depletion for GEM Propellant Aged in Pacific Ocean Seawater**

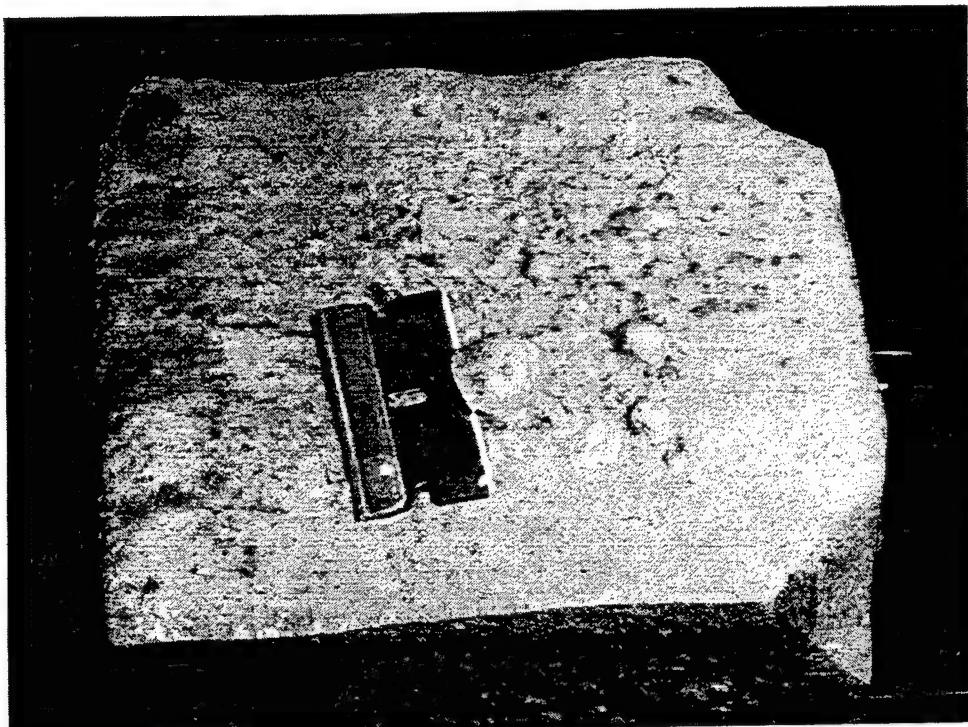
For reference 1-in. cubes were aged in deionized water for a period of up to 32 weeks. Figure 6 shows that their data falls on the same line as data for the samples aged in Pacific Ocean seawater whether or not the samples were sand buried. There is no ready explanation for these results.

Last September blister-like raised areas began to appear on some of the 2- and 4-in. cubes (Fig. 7). They did not appear on either the 1-in. or 15-in. cubes. No ready explanation was available for this blister phenomenon. It was initially thought that blister bubbles formed on propellant surfaces when aluminum in the propellant began to react with seawater producing hydrogen gas. Hypodermic syringes removed small gas samples from a few blister bubbles. Gas chromatographic analysis of the gas samples revealed nitrogen, oxygen and carbon dioxide. Thus, evidence for hydrogen production was absent. Since air contamination was probable, only carbon dioxide could be positively identified as coming from the propellant samples. The cause of carbon dioxide formation remains unexplained. Later, a large blister was cut off of a propellant cube surface with a razor blade for analysis. Before the blister was completely removed, the blister and its surrounding area were briefly examined. The blister back was solid and not any kind of film. Blister removal showed a water filled cavity. After removing the water from the cavity and enlarging the opening around the cavity, one could see that the exposed surface was identical to that of the bottom side of the removed blister. These events and observations are shown in Figures 8 to 11. Since these blisters usually occur over near surface propellant voids, it might be possible that this blister effect has a microbiological cause. An important observation was made that makes this explanation more probable. This past winter during a heating system power outage, pool temperatures dropped about 15 degrees for about 10 days. Propellant cube surface sliminess disappeared and there was an apparent reduction in blister population and size during this time period. Propellant cube surface sliminess reappeared once 75°F pool temperature was restored. Examination of Figure 6 shows that this brief temperature drop did not affect AP depletion results.

Dimensions were measured on some of the wet seawater aged propellant cubes. Resulting aging volumes for the different sized cubes were calculated and divided by their initial or unaged cube volumes. In turn, these values were plotted against seawater exposure time to show how sample swelling increased with time. These results were expected to parallel the oxidizer depletion rate data shown in Figure 6. A plot of the data can be seen in Figure 12. It should be pointed out that volume relationships contain relatively large errors because sample irregularities are magnified with swelling. Regardless of inherent errors in the data, the general trends observed in Figure 6 were also observed with respect to sample volume.



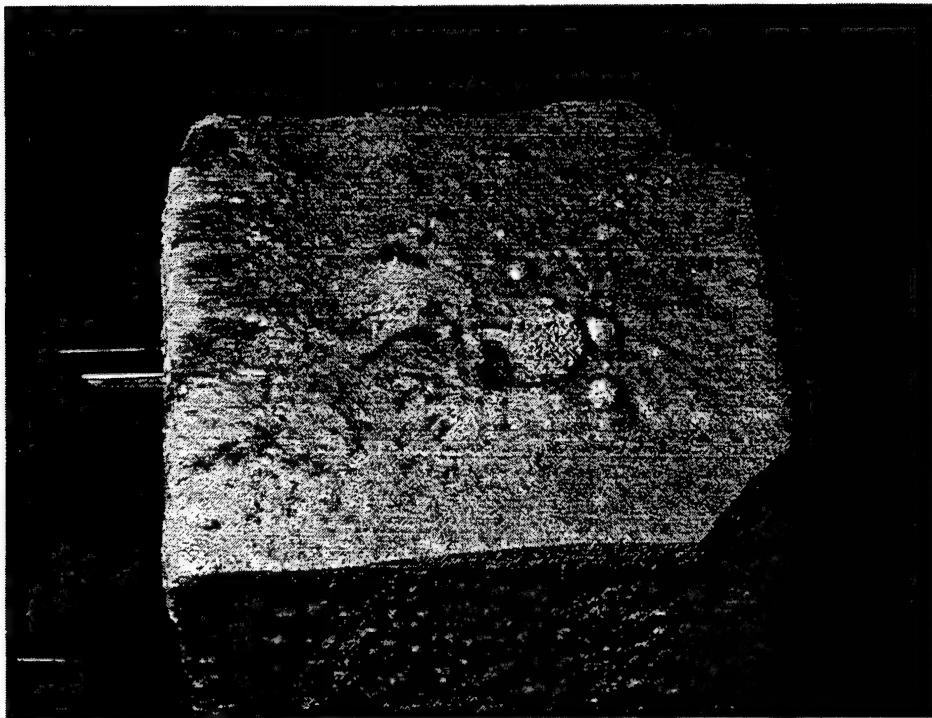
Figure 7. Appearance of Blisters on GEM Propellant Aging in Pacific Seawater



**Figure 8. Cutting into the Blister on GEM Propellant Cube Aged in Pacific Ocean Seawater**



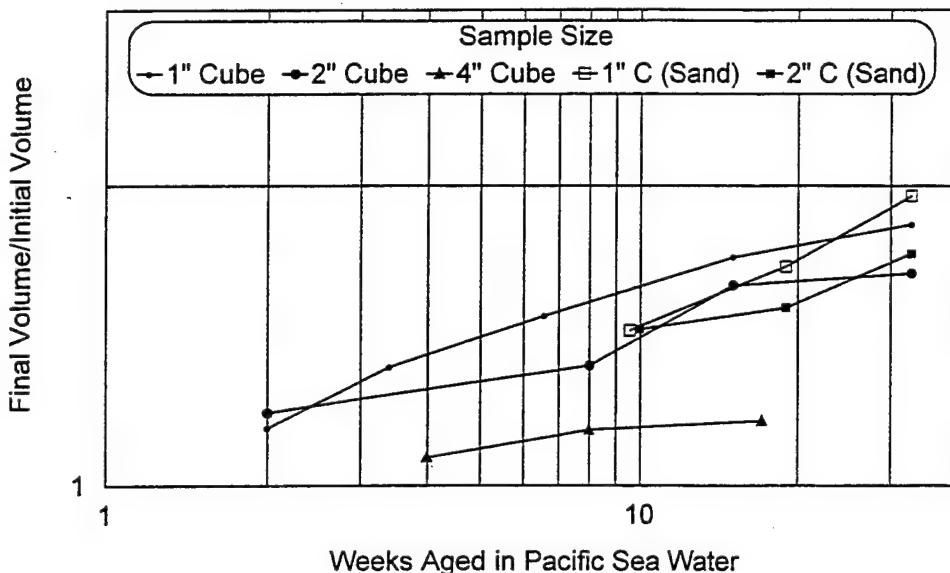
**Figure 9. Appearance of Blister on GEM Propellant Almost Completely Cut Through**



**Figure 10. Under Surface of Cutaway Blister on GEM Propellant  
Showing Underside Surface of Blister and Water  
Filled Void Beneath it**



**Figure 11. Water Removed from Void Underneath Cutoff Blister on  
GEM Propellant Showing its Surface**



**Figure 12. Relative Swelling Rates of Wet Aged GEM Propellant**

During drying of oxidizer depleted propellant cubes, white spots appeared on cube surfaces (Fig. 13). It was hypothesized that the spots were AP rather than sea salt. To check on this, Differential Scanning Calorimetry (DSC) thermograms were taken of the spots of a representative dried sample and some propellant grade AP. The results are shown in Figures 14 and 15. The essentially identical endothermic phase change curve shows that the spots were essentially AP.

Both impact and friction tests were run on some of the propellant cubes (dry and wet) that were submerged in the Pacific Ocean seawater. An Olin Mathieson Model 7 Drop Weight Tester was used for the impact tests. Test samples were 0.30-in. in diameter by about 20/1000-in. thick. A Julius Peters Model 21, Friction Tester was also used. Average friction test samples were ½-in. x ¼-in. x 20/1000-in. in dimension. Most tests were run on both the outside skin or surface, where AP depletion was greatest, and on the central interior. A non-aged control sample was also tested. All samples were distinguished and identified by cut corners (number and position), notches (number and position) or by a combination of cut corners and notches (number and position).

Available data on 1- and 2-in. cubes is presented in Table 3. Impact data shows that wet aged cubes are much less reactive and sensitive than their dried counterparts. This is especially true for outer skins or surfaces as illustrated by Samples 0-7 and 2c-1i in Table 3 with impact values of 102 kg-cm and 250 kg-cm, respectively. Even the centers of the wet cubes were less sensitive than the centers of the dry cubes (1-in. Samples 4ccc and 6c, respectively). It should be noted that the centers of 2-in. cubes were more sensitive than the 1-in. cubes' centers, (e.g., 108 kg-cm for 2-in. cube, Sample 8 versus 132 kg-cm for 1-in. cube, Sample 2c-1i). This is not surprising since more AP has leached out of the 1-in. cubes at any given time because of their more efficient surface-area-to-weight ratio. It is important to note, that after about 50% of the AP has leached out of the 1-in. cubes and the samples dried, the surface skin (Sample 0-5) is almost as insensitive as the surfaces of any aged wet sample. Friction data shows that the outer surface of the wet aged cubes is much less sensitive than their centers as is illustrated by 1-in. cube data for Sample 2c-1i. The sensitivity of wet sample centers remains similar to the sensitivity of dried samples as illustrated by Samples 2c-1i and Reference. Friction data, like the impact data, shows that after about 50% of the AP has been leached out of the 1-in. cubes and then dried, the surface skin is now as

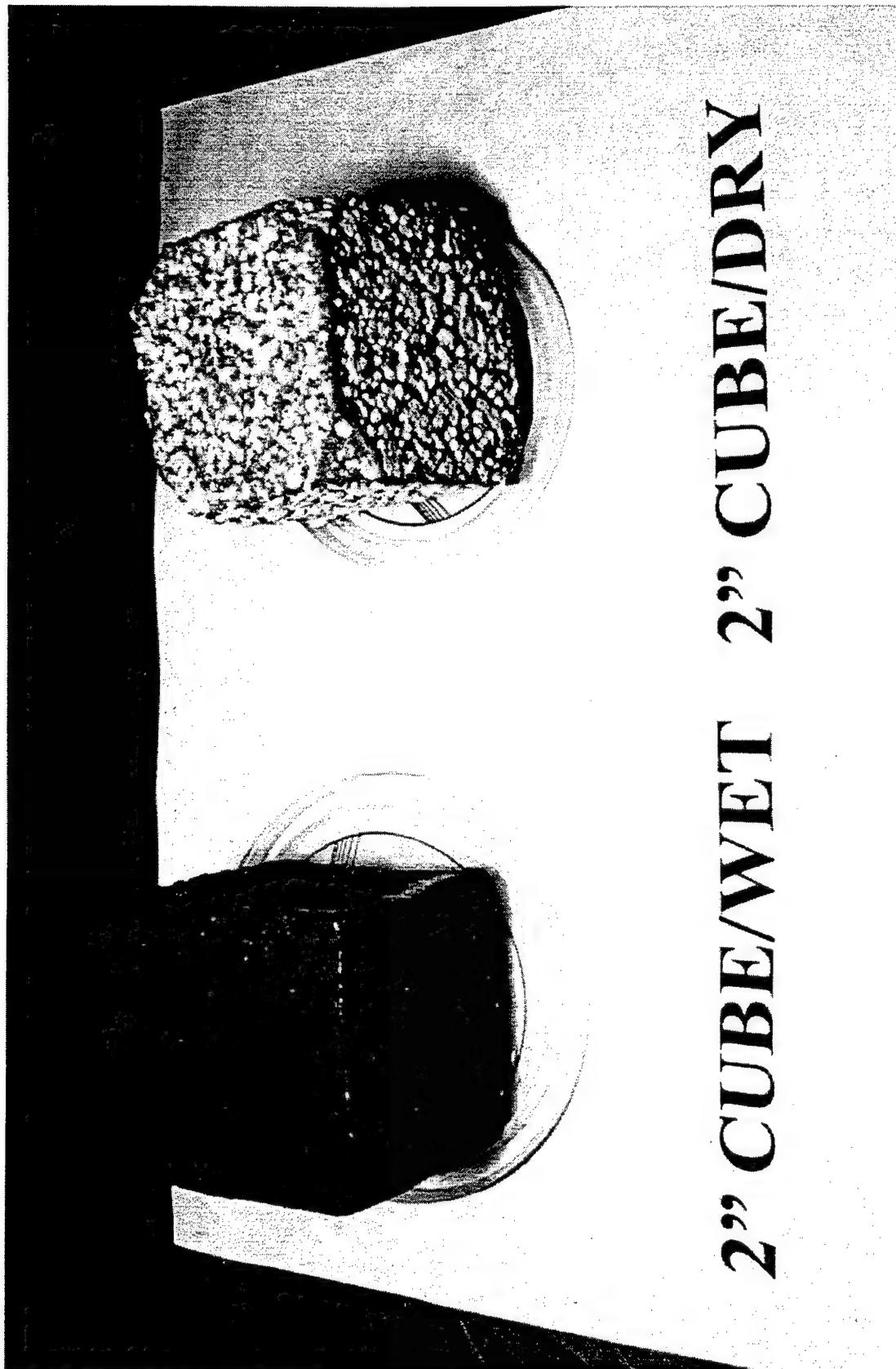
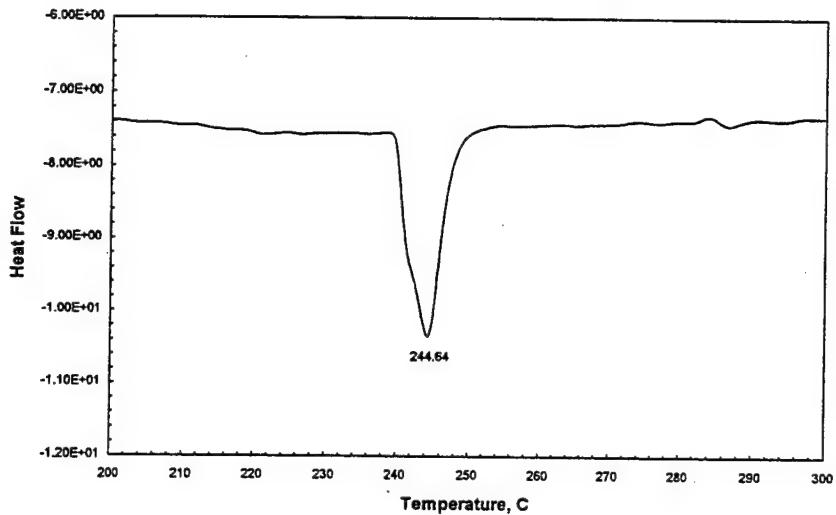
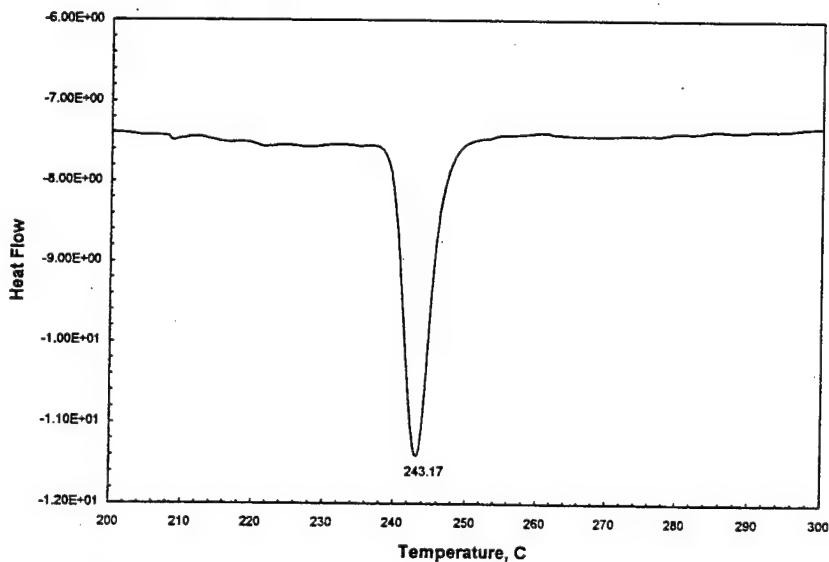


Figure 13. Representative Appearance of Wet and Dry GEM Propellant Cubes that Were Aged in Pacific Seawater



**Figure 14. Differential Scanning Calorimeter (DSC) Thermogram of Ammonium Perchlorate (AP)**



**Figure 15. Differential Scanning Calorimeter (DSC) Thermogram of White Crystalline Spots Located on Surfaces of Dried Cubes**

insensitive as the surfaces of any aged wet sample. All wet, immersion aged cubes had wet interiors. This was determined by comparison with corresponding dried samples.

Fire tests were run for both wet and dry propellant cubes that had been submerged in seawater. A dry control sample was also tested. Test results are presented in Tables 4 and 5. The seawater soaked, but dry, samples behaved very much like dry, untreated propellant. The dried cubes rapidly ignited. Once ignited, the flame front

**Table 3. Friction and Impact Test Results of Aged GEM Propellant**

Sample	Cube Size	Aging Time <sup>a</sup>	No Fires <sup>e</sup>	Location/State	Impact, kg-cm	Frict.,E,kg-cm
Reference <sup>b</sup>	-	0.00	4 (7)	Dry	93	12.0
0-7	1"	2.00	5 (5)	Skin/Dry	102	9.6
2c-1i	1"	2.00	5 (5)	Skin/Wet	250 <sup>c</sup>	28.8
-	1"	2.00	5 (5)	Center/Wet	132	14.4
3c-2io	1"	3.43	5 (5)	Skin/Dry	108	16.8
3c-3io	1"	3.43	5 (5)	Skin/Wet	250 <sup>c</sup>	36.0 <sup>d</sup>
-	1"	3.43	3 (5)	Center/Wet	240	21.6
6c	1"	6.97	4 (5)	Skin/Dry	99	14.4
-	1"	6.97	3 (5)	Centre/Dry	96	-
4ccc	1"	6.97	5 (5)	Skin/Wet	250 <sup>c</sup>	36.0 <sup>d</sup>
-	1"	6.97	5 (5)	Center/Wet	141	21.6
2i	1"	9.97	3 (5)	Skin/Dry	96	14.4
-	1"	9.97	4 (5)	Center/Dry	99	-
3c-3io	1"	9.97	5 (5)	Skin/Wet	250 <sup>c</sup>	36.0 <sup>d</sup>
-	1"	9.97	5 (7)	Center/Wet	138	28.8
0-5	1"	43.43	2(4)	Skin/Dry	200	36.0 <sup>d</sup>
0-6c	1"	43.43	5(5)	Skin/Wet	250 <sup>c</sup>	36.0 <sup>d</sup>
0-2c	1"	53.12	7(7)	Skin/Dry	250 <sup>c</sup>	36.0 <sup>d</sup>
0-4cccd	1"	53.12	5(5)	Skin/Wet	250 <sup>c</sup>	36.0 <sup>d</sup>
8	2"	2.00	5 (5)	Skin/Wet	250 <sup>c</sup>	32.4
-	2"	2.00	4 (5)	Center/Wet	108	14.4
0-5cccd	2"	4.00	5 (5)	Skin/Wet	250 <sup>c</sup>	36.0 <sup>d</sup>
-	2"	4.00	4 (5)	Center/Wet	105	14.4
1	2"	8.00	5 (5)	Skin/Wet	250 <sup>c</sup>	32.4
-	2"	8.00	4 (5)	Center/Wet	105	14.4
3cc-2ii	2"	9.97	5 (5)	Skin/Wet	250 <sup>c</sup>	36.0 <sup>d</sup>
-	2"	9.97	4 (5)	Center/Wet	114	14.4
0-6c	2"	43.43	5(5)	Skin/Wet	250 <sup>c</sup>	36.0 <sup>d</sup>
4cccd	2"	53.12	5(5)	Skin/Wet	250 <sup>c</sup>	36.0 <sup>d</sup>

a. Aging time in weeks

d. 36.0 kg-cm friction values is upper limit for test.

b. Propellant was not aged.

e. Number of samples out of the total number of samples,

c. 250 kg-cm impact value is upper

which is in parenthesis, that did not react or

limit for test.

decompose when impacted by a 2 kg weight.

quickly spread over the entire surface of the cubes. After about 15 weeks, the dried, aged 1-in cubes experienced a slight ignition delay. After 15 weeks, the longer these cubes were aged, the greater the ignition delay they experienced coupled with flame fronts spreading more slowly over all of their exposed surfaces. A test sample showed that AP loss was sufficient to leave post test skeletal remains of the samples. In other words, enough AP had been leached out of the test samples for a portion of the sample to act as though it was an insulator. This is shown in Figure 16.

The wet aged cubes were different. They required longer heating times before they could sustain combustion. This worked well where isoctane was used as the fuel for the fire test until a critical amount of AP had been leached from the samples. At that point, combustion could not be sustained. This varied for the 1-, 2-, 4- and 15-in. cube samples. The 1-in. cubes could not sustain burning after more than 19 weeks and less than 33 weeks. At this point, the test fuel was changed from isoctane to isopropanol. Isopropanol burned less vigorously and with a less smoky flame. Although this new fuel did not work for the oldest cubes, for a while it was successful with the 2-in and 4-in. cube samples. The next modification to improve burning conditions during the fire test was to burn a dry cube adjacent (about 1/4-in.) to a wet cube where both cubes had been aged for the same length of time. Burning the dry cube adjacent to the wet one would facilitate burning of the wet cube by reducing its surface moisture. It worked temporarily with the 2-in. cubes. Compare Test 54 with Test 56. When dry and wet cubes

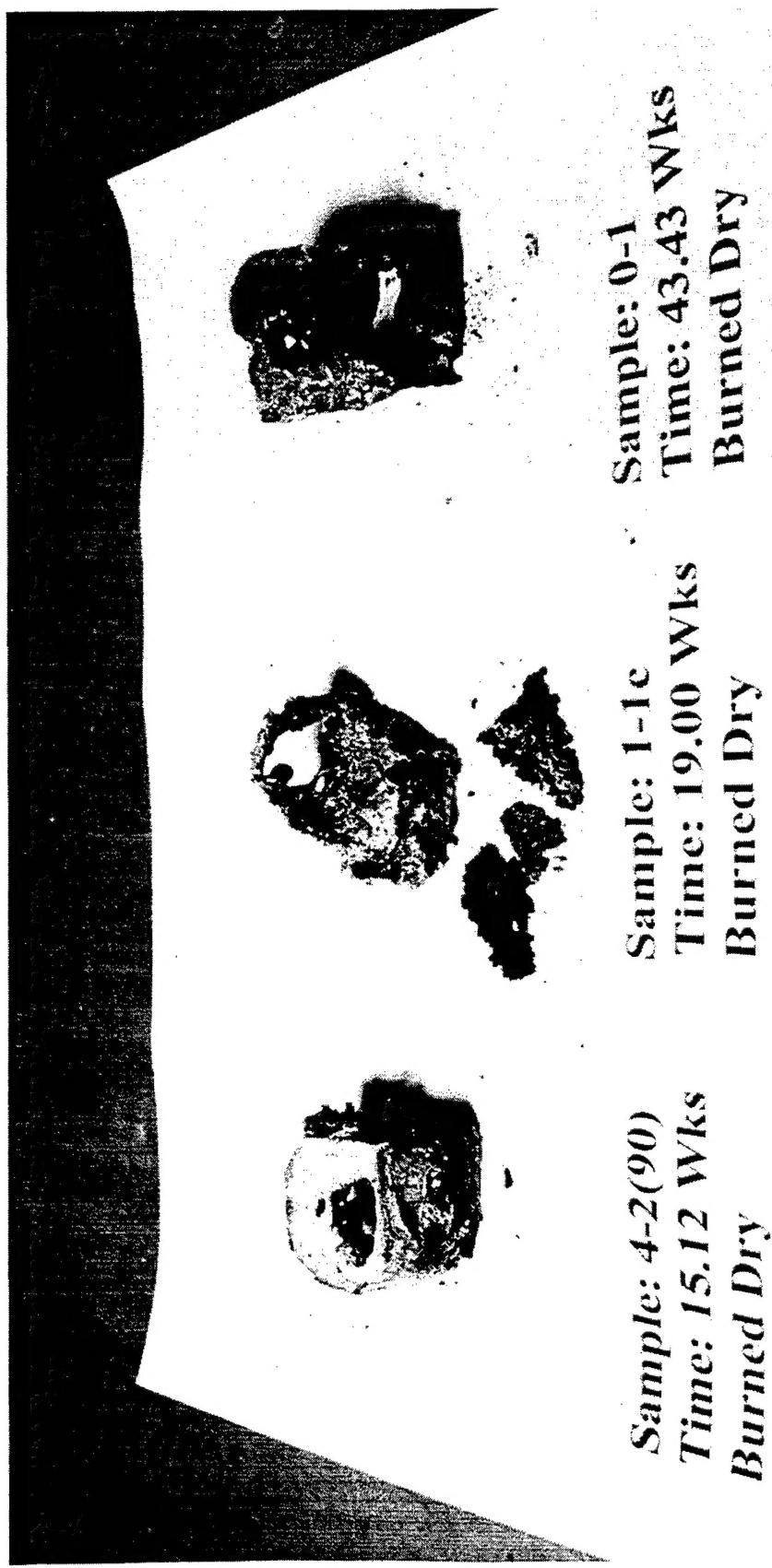


Figure 16. Post-Fire Test Results on 1-in. GEM Propellant Cubes that Were Aged from 15.12 to 43.43 Weeks in Pacific Seawater

**Table 4. Effect of Aging Time and Sample Size and State on Fire Test Results for Tests 1-35**

Test	Sample	Aging Water	Cube/State	Soak Time <sup>a</sup>	Time to Burn <sup>b</sup>
1	Control	None	1"/Dry	0.00	0
2	5-5i	S <sup>c</sup>	1"/Wet	1.11	60
3	7	S	1"/Dry	1.11	0
7	2-1i	S	1"/Dry	2.97	0
8	1-1o	S	1"/Wet	2.97	125
15	1-1i	S	1"/Dry	5.12	0
16	3-1i	S	1"/Wet	5.12	225
11	6t	S	2"/Dry	2.08	0
12	3	S	2"/Wet	2.08	90
17	2c	S	2"/Dry	5.12	0
18	1	S	2"/Wet	5.12	>300 <sup>f</sup>
25	2t	S	2"/Wet	5.12	200
26	5	S	2"/Dry	8.69	0
27	0	S	2"/Wet	8.69	270
4	3	C <sup>d</sup>	1"/Wet	1.11	55
5	0	C	1"/Dry	2.97	0
6	5	C	1"/Wet	2.97	115
9	3cc	P <sup>e</sup>	1"/Wet	2.00	225
10	3cd	P	1"/Dry	2.00	0
19	2c-2i0	P	1"/Dry	3.43	0
20	3c-1ii	P	1"/Wet	3.43	140
28	4cd	P	1"/Dry	6.97	0
29	5 3(90)	P	1"/Wet	6.97	425
32	2t-3iid	P	1"/Dry	9.97	0
33	0	P	1"/Wet	9.97	348 <sup>g</sup>
13	6t	P	2"/Dry	2.00	0
14	6c	P	2"/Wet	2.00	60
21	3cc-1i	P	2"/Dry	4.00	0
22	2c-2i0	P	2"/Wet	4.00	120
30	2c	P	2"/Dry	8.00	0
31	0-2c	P	2"/Wet	8.00	352
34	3cc-200	P	2"/Dry	9.97	0
35	4ccc-1op	P	2"/Wet	9.97	354
23	3tt	P	4"/Dry	4.00	0
24	6t	P	4"/Wet	4.00	120

a. Time in weeks

b. Approximate time in seconds

c. Simulated seawater

d. Cape Canaveral water

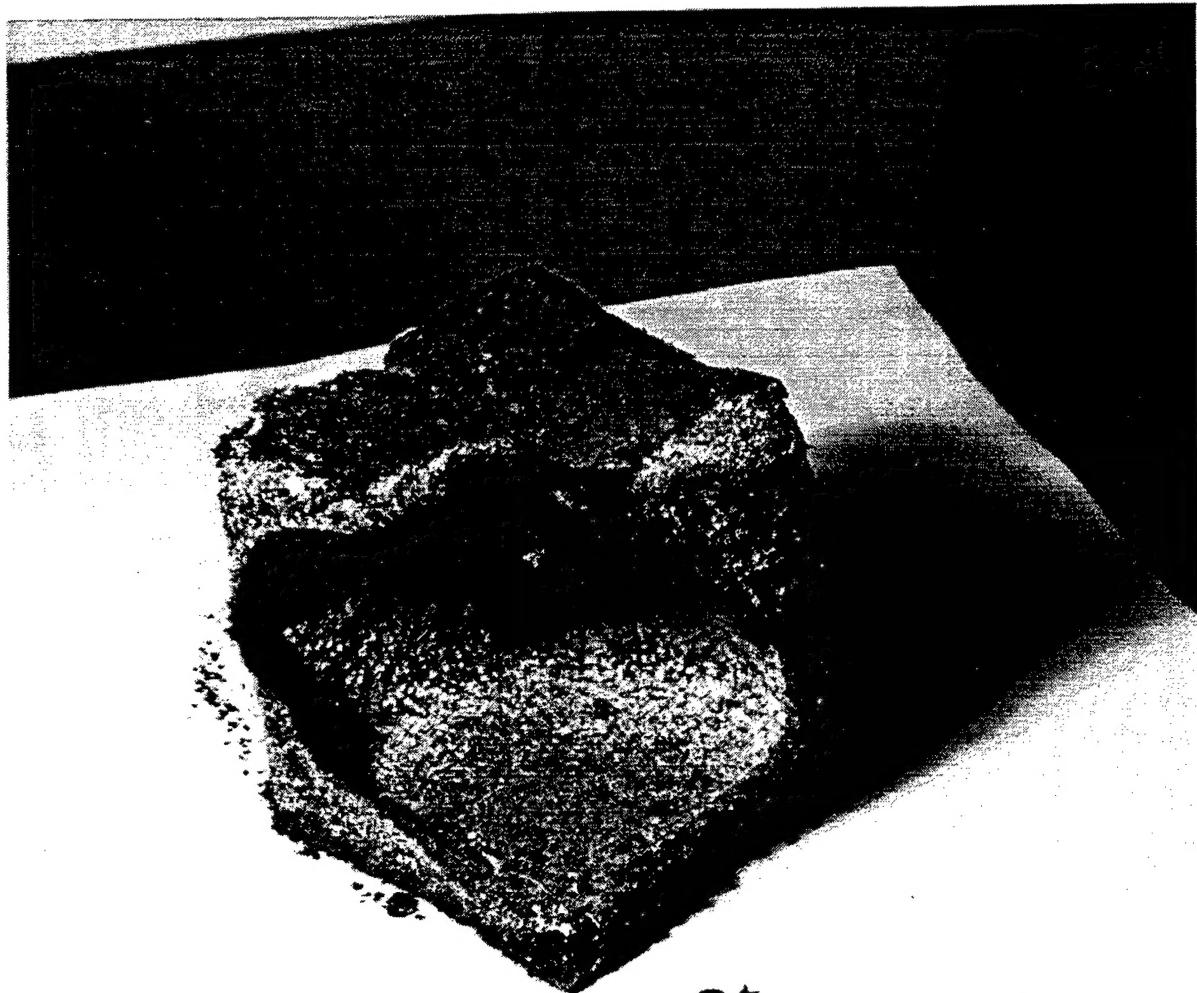
e. Pacific seawater

f. Sample not burned before fuel flame

expiration.

g. Fire intensity greater than Test 29

were burned adjacent to one another, it still took a long time before the wet cube finally sustained combustion. Figure 17 shows the shell left by the wet cube from Test 54. The shell did not char. Note the white spots on the surfaces. The shell was soft when pressed with the fingers. Nevertheless, once sustained combustion was obtained for the wet cubes, only one face of the cube burned. This might be linked to the fire resistance of AP-poor surfaces with good insulating properties that reduce heat flux to the propellant's interior. Propellant sample wetness throughout and external flame asymmetry probably influenced the tendency for asymmetric burning. In general, burning delay was directly related to sample size, water exposure or AP loss. Hence, the greater the AP loss from a wet cube, the longer and more difficult it became to maintain sustained combustion. Fire test videos show an interesting phenomenon related to the wet water soaked samples. Before sustained wet propellant burning occurred, transitory burning of numerous small to medium surface spots was observed. There were times when it was initially difficult to decide whether or not the propellant burning was transitory or sustained.



Sample: 2t  
Time: 43.43 Wks.  
Burned Wet &  
Adjacent to 3t

**Figure 17. Shell Left by a 2-in. Wet GEM Propellant Cube Aged for 43.43 Weeks in Pacific Seawater After a Fire Test in which It Was Adjacent to a 2-in. Dry Cube Aged for the Same Period**